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(54) Title: PROCESS FOR REMOVING SULFUR AS A LIQUID DURING GAS TREATING		
(57) Abstract <p>A process for removing liquid sulfur from a reaction mixture during a gas treating process, especially a process for removing hydrogen sulfide from a gaseous stream. Sulfur which is formed during the process is removed from a reaction mixture by first concentrating the sulfur in a small portion of the reaction mixture and then heating this portion to a temperature which is sufficient to melt the sulfur. The molten sulfur is removed from the reaction mixture and washed with a suitable solvent to remove any remaining reaction mixture.</p>		

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5 **PROCESS FOR REMOVING SULFUR AS A LIQUID DURING GAS
 TREATING**

BACKGROUND OF THE INVENTION

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FIELD OF THE INVENTION:

 The present invention relates to a process for removing liquid sulfur from
a reaction mixture during a gas treating process, and more particularly, to a
process for removing hydrogen sulfide from a gaseous stream wherein sulfur is
15 removed from a reaction vessel as a slurry with a portion of the reaction mixture,
heated to its melting point, and washed to remove any remaining reaction mixture
therefrom.

DESCRIPTION OF RELATED ART:

 Numerous industrial processes, particularly those pertaining to the
20 petroleum industry, generate gaseous by-products containing hydrogen sulfide,
either alone or in a mixture with water and/or other gases, such as, methane,
carbon dioxide, low molecular weight hydrocarbons, nitrogen, ammonia etc. In
addition, natural gas which is produced from subterranean formations often
contains similar gases to those gaseous by-products listed above. For many
25 years, these gaseous by-products were oxidized by common oxidation processes,
such as the Claus process, to obtain sulfur. In accordance with the Claus
process, hydrogen sulfide is oxidized by direct contact with air to produce sulfur
and water. However, several disadvantages of air oxidation of hydrogen sulfide,
including loss of a valuable hydrogen source, precise air rate control, removal of
30 trace sulfur compounds from spent air, and an upper limit on the ratio of carbon
dioxide to hydrogen sulfide, led to the development of alternative processes for
the conversion of hydrogen sulfide in gaseous by-products to sulfur.

 As detailed in U. S. Patent Nos. 4,592,905 and 5,334,363 to Plummer et
al., one such alternative process involves contacting within a reactor a feed gas
35 containing hydrogen sulfide with an anthraquinone which is dissolved in a polar
organic solvent. This polar organic solvent preferably has a polarity greater than
about 3 Debye units. The resulting reaction between hydrogen sulfide and
anthraquinone yields sulfur and the corresponding anthrahydroquinone. The

sulfur precipitates from the solution in crystalline form and is recovered as a product while the remaining solution containing anthrahydroquinone is thermally or catalytically regenerated producing the initial anthraquinone form and releasing hydrogen gas. The anthraquinone is recycled back to the reactor and the hydrogen gas is recovered as a product. As just mentioned, the insoluble sulfur, e.g. S₈ or other forms of polymerized sulfur, is conventionally withdrawn from the reactor as a precipitate in the reaction solution, is separated from solution by filtration, centrifugation or other means known in the art, is washed to remove the polar organic solvent, dissolved anthrahydroquinone, any unreacted anthraquinone and complexing agent, and is dried or melted to a liquid form. For applications, such as gas plants, where sulfur production is relatively small, a significant disadvantage of this process is that the removal of sulfur as a solid with subsequent washing to obtain maximum purity is expensive due to the filtration and centrifuge equipment which is required. To date, however, removal of sulfur as a solid has been more practical than removal of sulfur as a liquid from the reaction solution. Removal of sulfur as a liquid from the liquid reaction mixture is problematic since sulfur dissolves in the reaction solution above about 30° C. and melts at from about 112° C. to about 118° C. where significant reaction of sulfur and anthrahydroquinone to hydrogen sulfide and anthraquinone occurs. Thus, a need exists for a process for removing hydrogen sulfide from a gaseous feed by contacting within a reactor the feed gas containing hydrogen sulfide with a quinone which is dissolved in a polar organic solvent wherein sulfur which is formed from the reaction between hydrogen sulfide and quinone is removed as a liquid from the reaction solution.

Accordingly, it is an object of the present invention to provide a process for removing sulfur as a liquid from a reaction solution during the treatment of a gaseous stream to remove hydrogen sulfide therefrom.

It is another object of the present invention to provide a process for removing sulfur as a liquid from a reaction solution during the treatment of a gaseous stream to remove hydrogen sulfide therefrom which is relatively inexpensive.

It is a further object of the present invention to provide a process for washing liquid sulfur to remove any remaining reaction mixture from the liquid sulfur which has been previously separated from the reaction solution during the treatment of a gaseous stream to remove hydrogen sulfide therefrom.

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SUMMARY OF THE INVENTION

To achieve the foregoing and other objects, and in accordance with the purposes of the present invention, as embodied and broadly described herein, one characterization of the present invention comprises a process for converting
10 hydrogen sulfide to sulfur. A feed gas containing hydrogen sulfide is contacted with a polar organic solvent having a quinone dissolved therein. The hydrogen sulfide reacts with the quinone to produce sulfur and a corresponding hydroquinone in the solvent. Sulfur is concentrated in a portion of the polar organic solvent which is heated to a temperature sufficient to melt the sulfur. The
15 molten sulfur is removed from the polar organic solvent.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention relates to the removal of liquid sulfur from a reaction mixture obtained during a process for the conversion of hydrogen sulfide which
20 is initially contained in a gaseous feed stream to elemental sulfur. In accordance with the process of the present invention, the gaseous feed containing hydrogen sulfide (H_2S) is contacted in a reactor with a polar organic solvent having a quinone dissolved therein. The polar organic solvent may also have a complexing agent dissolved therein. The solvent preferentially solubilizes hydrogen sulfide
25 from the feed gas to form a reaction solution which is maintained in the reactor at a temperature and a pressure, as hereinafter discussed, and for a time which is sufficient to convert the hydrogen sulfide and quinone to sulfur and the corresponding hydroquinone. The solvent also solubilizes significant portion of the water, low molecular weight hydrocarbons, i.e. $C_2 - C_8$, and/or carbon dioxide
30 present in the gaseous feed stream. Should the feed gas contain large quantities of such gases other than hydrogen sulfide which are inert to this process, i.e. nitrogen, carbon dioxide, ammonia, methane or other low molecular weight

hydrocarbons, it may be necessary to initially contact the feed gas and polar organic solvent containing a quinone in an absorber. In addition, the feed gas may contain other sulfur compounds, such as COS, CS, and mercaptans, which are dissolved in the polar organic solvent and converted in the process to H₂S, recycled to the reactor, and converted to sulfur. The absorber and reactor may be separate components of a single processing unit or a single reaction vessel, for example a stirred tank, in which both functions are performed.

The polar organic solvent utilized in the process of the present invention is chosen to have a high polarity, i.e. greater than about 3.0 Debye units, and yet remain stable at dehydrogenation temperatures. Suitable polar organic solvents include N-methyl-2-pyrrolidinone, N,N-dimethylacetamide, N,N-dimethylformamide, sulfolane (tetrahydrothiophene-1,1-dioxide), acetonitrile, 2-nitropropane, propylene carbonate and mixtures thereof. The most preferred solvent is N-methyl-2-pyrrolidinone (NMP).

In accordance with the present invention, a complexing agent may also be incorporated into the polar organic solvent. This complexing agent is believed to react with H₂S to form an ion complex (CAH⁺HS⁻). Use of a complexing agent of high basicity results in increased ion complex formation and therefor increased conversion of quinone. In accordance with the present invention, the pK_a of the complexing agent is less than about 13.0, more preferably less than about 9.0, and most preferably less than about 6.0. The pK_a values are based on K_a (equilibrium constant) of 14.0 for the dissociation of water and are measured at 25° C. Suitable complexing agents are selected from amines, amides, ureas, nitrogen containing heterocyclic aromatics, guanidines, imidazoles, and mixtures thereof. These complexing agents can also be substituted with alkyl, aryl and organic alcohol groups. Examples of suitable complexing agents are n-methylacetamide, pyridine, substituted pyridines, diethylmethylanine, tributylamine, methyldiethanolamine and tetramethylurea. The preferred complexing agents are diethylmethylanine (DEMA), methyldiethanolamine (MDEA), tributylamine (TBA), pyridine (PY), and substituted pyridines. The molar ratio of complexing agent to quinone in the polar organic solvent is about 1:50 to about 2:1 and preferably about 1:6 to about 1:1.

In general, the quinone utilized in the process of the present invention is selected from anthraquinones, benzoquinones, naphthaquinones, and mixtures thereof and are chosen to maximize its reaction with H_2S . Choice of the quinone is based on such properties as the solubility of quinone in the polar organic solvent. Solubility is a function of the groups substituted on the quinone. For example, alkyl quinones have much higher solubilities than sulfonated quinones. Useful alkylquinones are ethyl, t-butyl, t-amyl and s-amyl quinones and mixtures thereof because of their relatively high solubilities in most polar organic solvents.

Preferably, the reaction solution, i.e. the polar organic solvent having a suitable quinone, complexing agent and hydrogen sulfide dissolved therein, is maintained in the H_2S reactor at a temperature of from about $0^\circ C$. to about $70^\circ C$. and at a H_2S partial pressure of from about 0.05 to about 4.0 atmospheres.

The treated feed gas is removed from the reactor and transported for further treatment or for use. In accordance with the present invention, the insoluble sulfur, e.g. S_8 or other forms of polymerized sulfur, is withdrawn from the reactor as a precipitate in the reaction solution and is allowed to separate from a significant portion, i.e. about 90 to 95 vol %, of the reaction solution by settling at a temperature of about $0^\circ C$. to about $30^\circ C$. The insoluble sulfur is then removed from the vessel in which such separation occurs as a slurry with the remaining, about 5 to 10 vol%, reaction solution and is transported and introduced into a melter at a point immediately above any melted sulfur present therein. In the melter, the slurry is heated to a temperature slightly above the $112^\circ C$. - $118^\circ C$. melting point of insoluble sulfur, i.e. S_8 , by any suitable means, for example by using steam in an internal heat exchanger which is positioned within the liquid sulfur phase present in the melter. Any hydroquinone which is present in the slurry will be converted to quinone and hydrogen sulfide upon reaction with S_8 . The reaction solution and the liquid sulfur are separately removed from the melter. The liquid sulfur is washed with a suitable solvent, for example water, hydrocarbons or oxygenated hydrocarbons, such as, acetone, 3-pentanone, or mixtures thereof, to dissolve any remaining reaction solution from the liquid sulfur. This wash solvent has a boiling point of at least about $50^\circ C$, preferably at least about $100^\circ C$, and most preferably at least about $150^\circ C$ below that of the polar

organic solvent. The pressure on the wash solvent and the reaction solution is reduced to fractionate the solvent from the dissolved reaction mixture and the wash solvent is recycled back to the washing step. If water is utilized as the wash solvent, the water is recycled back to the reactor where it is dissolved in the polar organic solvent.

The reaction solution which is removed from the melter is combined with the reaction solution from which the sulfur slurry was previously separated. The combined reaction solution which contains hydroquinone, any unreacted quinone, complexing agent, solvent, and unreacted compounds from the feed gas, such as hydrogen sulfide, COS, CS₂, carbon dioxide, mercaptans, and unrecovered sulfur, is maintained at a temperature of from about 70° C. to about 220° C. and at a pressure of from about 0.01 to about 4.0 atmospheres to convert COS, CS₂, mercaptans and unrecovered sulfur to hydrogen sulfide. The reaction solution is then fed to a flash tank or fractionator where substantially all unreacted feed gas constituents, including water, CO₂ and/or low molecular weight hydrocarbons, are removed from solution, separated and recovered as products. The remaining gas which consists primarily of unreacted H₂S is recycled to the reactor. The solution is withdrawn from the flash tank or fractionator and is further processed to convert the hydroquinone to the corresponding quinone and products, such as hydrogen, hydrogen peroxide or water.

In accordance with one such example of further processing, the reaction solution further heated to from about 220° C. to about 350° C. at a pressure sufficient to prevent solvent boiling. The heated solution is then fed to a dehydrogenation reactor where the hydroquinone is catalytically or thermally converted to the initial quinone and hydrogen gas under the temperature and pressure conditions stated above. The presence of a complexing agent in the reaction solution fed to the dehydrogenation reactor results in an unexpected and marked increase in the selectivity of hydroquinone to quinone and hydrogen product, and thus, the attendant decrease in unwanted hydrogenolysis by-products. Quinone is withdrawn from the dehydrogenation reactor dissolved with the complexing agent in the polar organic solvent and is recycled to the H₂S-quinone reactor, while the hydrogen gas is recovered as a commercial product.

The following example demonstrates the practice and utility of the present invention, but are not to be construed as limiting the scope thereof.

Example 1

Fifty (50) gms of sulfur and 50 gms of a reaction mixture containing 22.80
5 wt% t-butyl anthraquinone (TBAQ) and t-butyl anthrahydroquinone (H_2 TBAQ),
68.40 wt% n-methyl-2-pyrrolidinone (NMP), 6.8 wt% pyridine (PY) and 1.9 wt%
water are slurried and heated to 123-133° C. to melt the sulfur. A 50.06 liquid
sulfur phase which contains 49.33 gms of sulfur and 0.73 gms of reaction mixture
is then removed. The 0.67 gm balance of sulfur is converted to hydrogen sulfide
10 upon reaction with the H_2 TBAQ.

The 50.06 gms of liquid sulfur phase is slurried with 10.00 gm of liquid
acetone at 123 -133° C. and 10 atmospheres pressure to keep the mixture as a
liquid phase. Essentially all of the 0.73 gms of reaction mixture is extracted into
the liquid acetone. The slurry is then separated and one essentially pure liquid
15 sulfur product is withdrawn from the separator. The acetone and reaction mixture
phase is also removed from the separator and fractionated at 2 atmospheres to
remove essentially all of the acetone as vapor from the liquid reaction mixture.
The liquid reaction mixture is mixed with the 49.27 gms of starting reaction
mixture. The acetone is pressurized back to 10 atmospheres to form a liquid
20 phase.

While the foregoing preferred embodiments of the invention have been
described and shown, it is understood that the alternatives and modifications,
such as those suggested and others, may be made thereto and fall within the
scope of the invention.

We claim:

- 1 1. A process for converting hydrogen sulfide to sulfur comprising:
 - 2 contacting a feed gas containing hydrogen sulfide with a polar
 - 3 organic solvent having a quinone dissolved therein, said hydrogen
 - 4 sulfide reacting with said quinone to produce sulfur and a
 - 5 hydroquinone in said solvent;
 - 6 concentrating said sulfur in a portion of said polar organic
 - 7 solvent; heating said portion of said solvent to a temperature
 - 8 sufficient to melt said sulfur; and
 - 9 removing said molten sulfur from said polar organic solvent.
- 1 2. The process of claim 1 wherein said portion of said polar organic solvent
- 2 is about 5 to 10 vol% of the total volume of said polar organic solvent with which
- 3 said hydrogen sulfide is contacted.
- 1 3. The process of claim 1 wherein said step of concentrating is performed
- 2 by allowing said sulfur to settle within said polar organic solvent.
- 1 4. The process of claim 3 wherein said sulfur is allowed to settle within
- 2 said polar organic solvent at a temperature of about 0° C. to about 30° C.
- 1 5. The process of claim 1 wherein said portion of said polar organic solvent
- 2 is transported and introduced into a melter at a point immediately above any
- 3 molten sulfur already present therein.
- 1 6. The process of claim 1 further comprising:
 - 2 washing the molten sulfur with a liquid solvent to dissolve any
 - 3 polar organic solvent from the molten sulfur.
- 1 7. The process of claim 6 wherein the solvent is water, a hydrocarbon or
- 2 an oxygenated hydrocarbon.
- 1 8. The process of claim 7 wherein the oxygenated hydrocarbon is acetone,
- 2 3-pentanone, or mixtures thereof.
- 1 9. The process of claim 6 wherein said solvent has a boiling point of at
- 2 least about 50° C below that of said polar organic solvent.
- 1 10. The process of claim 9 wherein said solvent has a boiling point of at
- 2 least about 100° C below that of said polar organic solvent.

1 11. The process of claim 10 wherein said solvent has a boiling point of at
2 least about 150° C below that of said polar organic solvent.

1 12. The process of claim 6 further comprising:
2 fractionating the wash solvent as a vapor from the polar organic
3 solvent.

1 13. The process of claim 12 wherein said solvent vapor is cooled to a liquid
2 and recycled to the washing step after being fractionated from the polar organic
3 solvent.

1 14. The process of claim 12 wherein water is the solvent and wherein the
2 water vapor is cooled to a liquid and recycled to the contacting step where said
3 water is dissolved in the polar organic solvent.

1 15. The process of claim 1 wherein said polar organic solvent has a
2 complexing agent dissolved therein.

1 16. The process of claim 15 wherein the pK_b value of said complexing
2 agent is less than about 13.0.

1 17. The process of claim 15 wherein said complexing agent is amines,
2 amides, ureas, nitrogen containing heterocyclic aromatics, guanidines, imidazoles,
3 or mixtures thereof.

1 18. The process of claim 17 wherein said complexing agent is substituted
2 with alkyl, aryl, or organic alcohol groups.

1 19. The process of claim 17 wherein said complexing agent is N-
2 methylacetamide, pyridine, substituted pyridines, diethylmethylamine,
3 tributylamine, tetramethylurea, or mixtures thereof.

1 20. The process of claim 1 wherein said polar organic solvent has a
2 polarity of greater than about 3.0 Debye units.

1 21. The process of claim 1 wherein said polar organic solvent is N,N-
2 dimethylacetamide, N-methyl-2-pyrrolidinone, N,N-dimethylformamide,
3 tetrahydrothiophene-1,1-dioxide, acetonitrile, 2-nitropropane, propylene carbonate
4 or mixtures thereof.

1 22. The process of claim 1 wherein said quinone is an anthraquinone,
2 benzoquinone, naphthaquinone, or mixtures thereof.

1 23. The process of claim 22 wherein said quinone is substituted with an
2 alkyl group.

1 24. The process of claim 23 wherein said alkyl group is ethyl, t-butyl, t-
2 amyl, s-amyl or mixtures thereof.

1 25. All inventions described herein.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US96/18727**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) : BO1D 53/52; CO1B 17/04, 17/05

US CL : 423/226, 242.2, 573.1, 576.2

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 423/226, 242.2, 243.01, 573.1, 576.2, 576.4

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
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NONE**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,334,363 A (PLUMMER) 02 August 1994, col. 3 line 15 - col. 4 line 63.	1 - 25
Y	US 4,304,570 A (KLEEGERG et al.) 08 December 1981, col. 1 lines 14-22 and col. 6 lines 11-37.	1
A	US 4,020,149 A (BOSNIACK) 26 April 1977, col. 8 lines 30-67.	1 - 25
A	US 4,069,302 A (MEADOW) 17 January 1978, col. 5 line 1 - col. 6 line 20.	1 - 25
A	US 4,174,383 A (DESCHAMPS et al.) 13 November 1979, col. 4 line 47 - col. 6 line 28.	1 - 25

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US96/18727

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	US 4,592,905 A (PLUMMER et al.) 03 June 1986, col. 7 line 4 - col. 8 line 48.	1 - 25
A	US 4,637,926 A (PHILLIPS et al.) 20 January 1987, col. 5 line 51 - col. 6 line 57.	1 - 25
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A	US 5,057,298 A (RAY et al.) 15 October 1991, col. 9 line 11 - col. 10 line 37.	1 - 25
A	EP 0 207 509 A2 (KLIEM et al.) 07 January 1987, please see the English abstract.	1 - 25
A	PLUMMER M. A. Gas Processing Developments: Sulfur and hydrogen from hydrogen sulfide, Hydrocarbon Processing, April 1987, pages 38 - 40.	1 - 25